

PATENT COOPERATION TREATY PCT

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INTERNATIONAL PRELIMINARY EXAMINATION REPORT (PCT Article 36 and Rule 70)

19 JAN 2005
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Applicant's or agent's file reference TS 9505 PCT	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA416)	
International application No. PCT/EP 03/08063	International filing date (day/month/year) 16.07.2003	Priority date (day/month/year) 19.07.2002
International Patent Classification (IPC) or both national classification and IPC C08L23/16		
Applicant SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 6 sheets, including this cover sheet.

☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

 These annexes consist of a total of 6 sheets.

3. This report contains indications relating to the following items:

I	<input checked="" type="checkbox"/>	Basis of the opinion
II	<input type="checkbox"/>	Priority
III	<input type="checkbox"/>	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
IV	<input type="checkbox"/>	Lack of unity of invention
V	<input checked="" type="checkbox"/>	Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
VI	<input type="checkbox"/>	Certain documents cited
VII	<input type="checkbox"/>	Certain defects in the international application
VIII	<input type="checkbox"/>	Certain observations on the international application

Date of submission of the demand 18.02.2004	Date of completion of this report 21.10.2004
Name and mailing address of the international preliminary examining authority: European Patent Office - P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Tx: 31 651 epo nl Fax: +31 70 340 - 3016	Authorized Officer Bergmans, K Telephone No. +31 70 340-4189

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. **PCT/EP 03/08063**

I. Basis of the report

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

Description, Pages

1, 5-17	as originally filed
2, 2a, 3, 4	received on 27.08.2004 with letter of 27.08.2004

Claims, Numbers

1-10	received on 27.08.2004 with letter of 27.08.2004
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2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)).

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

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EXAMINATION REPORT**

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**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability;
citations and explanations supporting such statement**

1. Statement

Novelty (N)	Yes: Claims	1-10
	No: Claims	
Inventive step (IS)	Yes: Claims	
	No: Claims	1-10
Industrial applicability (IA)	Yes: Claims	1-10
	No: Claims	

2. Citations and explanations

see separate sheet

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/EP 03/08063

Re Item I

Basis of the opinion

The amendments filed with the letter dated 27/08/2004 introduce subject-matter which extend beyond the content of the application as filed, contrary to Article 34(2)(b) PCT. The amendment concerned in claim 1 is the following: "... and the process oil. This amendment indicates that beside Fischer-Tropsch process oil one or more other process oil can be used. This amendment goes beyond the disclosure in the international application as filed, the report shall be established as if such amendment had not been made, and the report shall so indicate (Rule 70.2© PCT).

All other amendments are allowable under article 19(2)PCT

Clarity (Art. 6 PCT)

The claim 1 does not meet the requirements of Article 6 PCT. The statements "used of", "as obtained by" and composition comprising" indicates the suitability of the use-claim, the process-claim, and product-claim. Consequently, instead of limiting the scope of the claim, renders the category of the claim unclear (Guidelines C-III, 4.8a, 4.1 and Art. 84 EPC).

Novelty (Art. 33(2) PCT)

1. The document D1 (US4134870) discloses an elastomeric blend having improved processability (col. 2 l. 32) comprising an elastomeric polymer like EPDM terpolymers (claim 1 and 7) and a non-polar wax. The wax can be derived from various sources like Fischer-Tropsch (column 9 lines 8-10). In table 3 column 9 it is indicated that the paraffin wax has a kinematic viscosity of 8.2 at 99°C. The difference is the use of a Fischer-Tropsch process oil (present application) instead of a Fischer-Tropsch wax (document D1).
2. The document D2 (US4208310) discloses an elastomeric blend having good processability (col. 2 l. 37-43) comprising an elastomeric polymer like EPDM terpolymers (claim 1 and 2) and a non-polar wax. The wax can be derived from various sources like Fischer-Tropsch (column 7 line 29). In table 2 column 8 it is indicated that the paraffin wax has a kinematic viscosity of 8.2 at 99°C. In column 9 lines 29-40 it is indicated that the flash point of the wax is at least 350 °F(170°C), and the pour point is less than 40°F (4°C).

The difference is the use of a Fischer-Tropsch process oil (present application) instead of a Fischer-Tropsch wax (document D2).

3. The document D3 (EP0577255) discloses an elastomeric composition comprising an olefinic copolymer rubber, olefinic polymer, a mineral oil, and crystalline paraffin (claim 1). The olefinic copolymer rubber is ethylene-propylene diene (claims 3,4), the olefinic polymer is propylene or propylene copolymer (claim 6), and the crystalline paraffin is a Fischer-Tropsch wax (page 3 lines 13-21). The difference is the use of a Fischer-Tropsch process oil (present application) instead of a Fischer-Tropsch wax (document D3).

4. Document D4 (EP0776959) indicates the method of obtaining a Fischer-Tropsch processing oil by hydro-isomeration, separating and dewaxing (solvent and catalyst). The kinematic viscosity of the process oil described in document D4 is overlapping the kinematic viscosity in the application (page 3 column 3 lines 34-38). There is no disclosure found in the document indicating the use of the Fischer-Tropsch process oil in an EPDM composition.

5. The document D5 (US3102098) describes a Fischer-tropsch lubricating oil with a kinematic viscosity at 210 °F of above 10, a Flash point of above 500 °F, a pour point of -25 or -35 °F, and indicates evaporation losses (table 1 column 4). The oil can be used as a lubricating oil. There is no disclosure of using the Fischer-Tropsch process oil in an EPDM composition.

In view of the prior art cited, claims 1-10 appear to be novel and meet therefore the requirements of Art. 33(2) PCT.

Inventive step (Art. 33(3) PCT)

The document D2 discloses an elastomeric blend having good processability comprising an elastomeric polymer like EPDM terpolymers and a non-polar wax. The wax can be derived from various sources like Fischer-Tropsch. The composition further comprises a non-polar paraffinic process-oil characterised by a viscosity of 70-300 ssu, a flash point of least 350 °F, a pour point is less than 40°F.

The subject-matter of claims 1-10 differs from this known D2 in

A) the use of a Fischer Tropsch processing oil.

B) the composition further comprises a polyolefin component

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There is no evidence found in the present application showing that the distinguishable feature (A and B) leads to an effect (lower hydrocarbon emission in an EPDM composition).

The problem to be solved by the present invention may therefore be regarded as a further process oil.

The solution proposed in claims 1-8 of the present application is considered as inventive (Art. 33(3) PCT). There is no evidence found in the prior art indicating that the use of a Fisch-tropsch process oil in an EPDM composition would lead to a lower hydrocarbon emission.

The solution proposed in claims 9 and 10 of the present application cannot be considered as inventive (Art. 33(3) PCT). An elastomeric composition comprising an olefinic copolymer rubber, olefinic polymer (polyolefin component), a mineral oil, and crystalline paraffin is known from the prior art document D3.

27. 08. 2004

(43)

EP-A-577255 describes an EPDM composition which contain an extender oil and a crystalline paraffin wax. The crystalline wax may be prepared by a Fischer-Tropsch process. The paraffinic wax has a melting point of between 60 and 100 °C and is thus solid at room temperature.

Compositions as described above are often used in automotive applications, such as parts of the interior of the automobile. There is an increasing demand for low hydrocarbon emissions of an automobile. These hydrocarbon emissions are measured by keeping a complete car at an elevated temperature and detecting any hydrocarbon emissions. In view of this development there is an increasing demand for EPDM containing compositions having a low hydrocarbon emission.

The object of this invention is to provide an EPDM containing composition having a low hydrocarbon emission.

This object is achieved by the following use. Use of a process oil having a kinematic viscosity at 100 °C greater than 8 cSt and a pour point of below 10 °C as obtained by (a) hydrocracking / hydroisomerising a feed comprising a Fischer-Tropsch synthesis product, (b) isolating from the product of step (a) a process oil precursor fraction, (c) dewaxing the process oil precursor fraction obtained in step (b) to obtain the process oil, optionally after separating a lower boiling fraction from said dewaxed product, as component in a composition comprising a ethylene-propylene-diene rubber component and the process oil.

Applicants have found that a process oil as derived from a Fischer-Tropsch synthesis product can be simply obtained having properties which lower the hydrocarbon emissions of the finished EPDM comprising product. Some severely hydroprocessed or synthetic paraffinic process oils as described above may

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also achieve this lower hydrocarbon emission. A disadvantage of these products is that they are either very expensive because they have to be synthesized from lower olefins or by heavily hydroprocessing. Another advantage of the Fischer-Tropsch derived oils as compared to the heavily

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hydroprocessed oils is that the low temperature properties for the higher viscosity grade oils is much better making the Fischer-Tropsch derived oils more easy to handle in the process to make the EDPM containing product.

The Fischer-Tropsch derived oil preferably has a flash point according to ISO 2592 of above 240 °C and more preferably above 260 °C. The UV adsorption of the oil at 300 nm is preferably less than 1% and more preferably less than 0.6% according to ASTM D 2008-A1. The evaporation loss at 107 °C during 22 hours of the oil (according to ASTM D 972 is preferably less than 0.1 wt% and more preferably less than 0.05 wt%.

The kinematic viscosity at 100 °C of the oil will be resultant from the above requirements and will ~~usually~~ be above 8 cSt and ~~more~~ preferably above 9 cSt. The upper limit is not essential for the hydrocarbon emissions requirements. Nevertheless it is not advantageous to use too viscous oil for practical processing reasons. Preferably the upper limit will be around 30 cSt, preferably 25 cSt. The pour point of the process oil will be dependent on the viscosity grade used. Applicants have found a process involving especially a catalytic dewaxing step to prepare a Fischer-Tropsch process oil having pour points ranging from -40 °C for the less viscous grades to around 10 °C for the more viscous grades. This has been found very advantageous because the oil can be used in a liquid state at ambient conditions during the manufacturing of the EPDM comprising composition. Applicants further found that the Fischer-Tropsch derived oil preferably has a CN number as measured according to IEC 590 of between 15 and 30%. The oil is preferably liquid at 20 °C.

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The process oil is ~~preferably~~ prepared using the below process, by

(a) hydrocracking/hydroisomerising a feed comprising a Fischer-Tropsch derived feed,

5 (b) isolating from the product of step (a) a process oil precursor fraction,

(c) dewaxing the process oil precursor fraction obtained in step (b) to obtain the process oil, optionally after separating a lower boiling fraction from said dewaxed product.

10 The Fischer-Tropsch derived feed can be obtained by well-known processes, for example the so-called commercial Sasol process, the commercial Shell Middle Distillate Process or by the non-commercial Exxon process. These and other processes are for example
15 described in more detail in EP-A-776959, EP-A-668342, US-A-4943672, US-A-5059299, WO-A-9934917 and WO-A-9920720.

20 A preferred process to prepare the process oil having the desired flash point, evaporation loss and CN-number properties involves using a Fischer-Tropsch derived feed in step (a) which is characterized in that the weight ratio of compounds having at least 60 or more carbon
25 atoms and compounds having at least 30 carbon atoms in the Fischer-Tropsch derived feed is at least 0.2 and wherein at least 30 wt% of compounds in the Fischer-Tropsch product have at least 30 carbon atoms. More preferably the feed has at least 50 wt% and most
30 preferably at least 55 wt% of compounds having at least 30 carbon atoms. Furthermore the weight ratio of compounds having at least 60 or more carbon atoms and compounds having at least 30 carbon atoms of the Fischer-Tropsch derived feed is preferably at

EPO - DG 1

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A M E N D E D C L A I M S

(43)

1. Use of a process oil having a kinematic viscosity at 100 °C greater than 8 cSt and a pour point of below 10 °C as obtained by

(a) hydrocracking / hydroisomerising a feed comprising a Fischer-Tropsch synthesis product,

(b) isolating from the product of step (a) a process oil precursor fraction,

(c) dewaxing the process oil precursor fraction obtained in step (b) to obtain the process oil, optionally after separating a lower boiling fraction from said dewaxed product, as component in a composition comprising a ethylene-propylene-diene rubber component and the process oil.

2. Use of the process oil according to claim 1, wherein the process oil has a flash point of above 260 °C according to ISO 2592.

3. Use of the process oil according to any one of claims 1-2, wherein the UV adsorption of the process oil at 300 nm is less than 0.6% according to ASTM D 2008-A1.

4. Use of the process oil according to any one of claims 1-3, wherein the evaporation loss of the process oil at 107 °C during 22 hours is less than 0.05 wt% according to ASTM D 972.

5. Use of the process oil according to any one of claims 1-4, wherein the kinematic viscosity at 100 °C greater than 9 cSt.

6. Use of the process oil according to any one of claims 1-5, wherein step (c) is performed by solvent dewaxing.

7. Use of the process oil according to any one of claims 1-5, wherein step (c) is performed by catalytic dewaxing.

8. Use of the process oil according to any one of claims 1-7, wherein the conversion in step (a) is between 25 and 65 wt%.
9. Use of the process oil according to any one of claims 1-8, wherein the composition furthermore comprises a poly-olefin component.
10. Use of the process oil according to claim 9, wherein the poly-olefin is polypropylene

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